

## SOLID-PHASE EXTRACTION BY C-18 COLUMN 5.3.1

The C-18 SPE column is used for samples that will be analyzed by capillary column gas chromatography/mass spectrophotometry with selected ion monitoring using NWQL schedule 2010 for a broad spectrum of pesticides.<sup>8</sup> Detailed descriptions of the method and laboratory and field extraction procedures are found in Zaugg and others (1995). For C-18 SPE processing, obtain a precleaned Analytichem™ SPE column (500 mg) and the other supplies and equipment described in the spike kit available from the NWQL (table 5-7).

***Quality-control samples are required as an integral part of the sampling program.***

- ▶ Process an initial field blank and then after about every 10 to 20 samples.
  - Use pesticide-grade blank water (PBW, obtained from the laboratory).
  - Process the blank in the same manner as you process the environmental water sample.
- ▶ Process a field matrix spike about every 20 samples. When processing a field matrix spike:
  - Collect duplicate samples.
  - Use a 100- $\mu$ L micropipet to add the spike solution (mixture) to one of the duplicate samples. The concentration of spike solution can vary, depending on availability and the needs of the study (1 ng/ $\mu$ L concentration is commonly used at this time). Follow the instructions provided with the spike kit.
  - Add the surrogate to every spiked sample and an associated unspiked sample.
  - Record lot number and concentration of spike mixture on the NWQL Schedule 2010 Reporting Form (worksheet) (fig. 5-2).

<sup>8</sup>C-18 solid-phase extraction method is used for isolation and concentration of 41 pesticides and pesticide metabolites with concentrations of 4 mg/L or less in natural water samples (atrazine, alachlor, cyanazine, and metolachlor have upper concentration limits of 20 mg/L) (Zaugg and others, 1995).



***Prepare to process samples onsite using the C-18 SPE column:***

1. Cover a bench or table with a sheet of aluminum foil to make a clean work surface. Put on appropriate disposable, powderless gloves.
2. Collect and split samples using the appropriate procedures (NFM 4; NFM 5.1; Sandstrom and others, 1995). Filter the samples as instructed in section 5.2.2. Wear gloves (usually latex or nitrile) during sample collection and processing.
3. Set up the necessary equipment and supplies and assemble them on the clean work surface. Remove the aluminum foil wrapping from the precleaned equipment.
4. Record the sampling site information, the lot number and dry weight of the C-18 SPE column, and the surrogate solution identification number on the Schedule 2010 worksheet (fig. 5-2).
5. Change gloves.
6. Tare the weight of a clean amber glass 1-L sample bottle and a 1-L plastic beaker to the nearest gram using an analytical balance and record the weights on the Schedule 2010 worksheet.

***Extract the sample:***

Use the appropriate surrogate solution mixture supplied by the NWQL for the C-18 SPE method with each environmental sample.

**SAMPLE EXTRACTION SHOULD BE COMPLETED ONSITE, IF POSSIBLE. If onsite extraction is not possible, extract the sample within 4 calendar days of collecting the sample.**

1. Condition the SPE column:
  - a. Pipet 2 mL of pesticide-grade methanol into the C-18 SPE column and allow it to flow through the column by gravity. Collect the methanol rinse in a proper container for disposal.
  - b. Remove any excess methanol by rinsing approximately 2 mL of PBW, by gravity, through the column. The rinse water/methanol mixture must be disposed of according to local, State, or Federal regulations.

c. **Do not allow the SPE column to go dry once the conditioning has started.**

- If the column goes dry, repeat the conditioning process.
  - To keep the column from drying out once the conditioning has started, maintain water in the C-18 SPE column by replacing water that drained through the column. Alternatively, attach an on/off valve-to-column outlet to prevent complete draining before the sample is extracted.
2. Following the filtration instructions for general organic compounds (5.2.2.A or Sandstrom, 1995), pass about 1 L of sample through the a glass microfiber filter into the tared bottle, leaving about 2 cm of headspace.
  3. Weigh the filled bottle and record the weight on the worksheet (fig. 5-2).
  4. Add about 10 mL of methanol to the filtered sample using the bottle-top dispenser or a volumetric pipet. Weigh and record the sample-plus-methanol weight on the worksheet.
  5. Add the surrogate solution contained in the 2-mL amber screw-cap vial to the filtered sample as follows (refer to Spike Kit Instruction Manual for detailed information and instructions on use of a micropipet):
    - a. Withdraw the surrogate solution from the 2-mL amber screw-cap vial using a clean 100- $\mu$ L micropipet and a clean glass bore.
    - b. Insert the tip of the glass bore into the sample bottle below the surface of the sample, and depress the plunger to deliver the surrogate to the sample. (Tip the bottle on its side, if necessary, to reach below the surface of the sample with the glass bore.)
    - c. Keeping the plunger depressed, swirl pipetor in water several times and then withdraw the micropipet from the bottle. Release the plunger, then remove the used glass bore from the micropipet and discard properly.
    - d. Rinse the fluorocarbon polymer tip of the micropipet with methanol.
    - e. Add the field-matrix spike as dictated by the study's quality-assurance plan, as required.
    - f. Cap and swirl the sample to mix the sample + surrogate. (For spiked samples, mix sample + surrogate + spike solution.)
    - g. If a duplicate will be submitted for analysis, repeat steps 5a–f on the duplicate sample.

6. Extract the sample through the SPE column using a metering pump fitted with 3.18 mm (1/8 in.) fluorocarbon polymer tubing with appropriate connectors (Sandstrom, 1995; NFM 2).
  - a. Insert clean tubing from the inlet side of the pump into the sample bottle.
  - b. Turn on the pump, flush air from the tubing (be careful to minimize any sample discharge from the end of the tubing), and then attach the outlet side of the tubing to the small end of the SPE column.
  - c. Invert the SPE column to drain any remaining conditioning water left in the SPE column reservoir.
  - d. Begin extraction by pumping the sample through the column at a rate of 20 to 25 mL/min and collect the extracted water into the tared 1-L plastic beaker.
7. After the sample has been pumped through the SPE column, turn the pump off and disconnect the column.
8. Remove excess sample from the SPE column using a syringe with 10 to 20 mL of air to push excess sample into a plastic beaker.
9. Weigh the beaker containing the volume of sample extracted through the SPE column. Subtract the tare weight of the beaker from the weight of the beaker plus the extracted sample and record this weight on the worksheet.
10. Write the sample identification number and the sampling date and time on the side of the SPE column. Place the SPE column into a 40-mL glass or plastic shipping ampoule and wrap it in aluminum foil.
11. Finish filling out the worksheet (fig. 5-2). Wrap the completed worksheet around the shipping ampoule and secure it with a rubber band or tape. Place in a sealable plastic bag.
12. Chill the SPE column immediately and maintain between 4°C and 25°C during storage and shipping.
13. Keep a copy of the worksheet for the field folder.

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14. Field clean all equipment, including the pump and tubing, immediately after use and before going to the next site (NFM 3).
  - a. Rinse thoroughly with about 50 mL of a 0.2-percent solution of a phosphate-free laboratory detergent, followed by about 50 mL of tap water (or DIW) to remove the detergent.
  - b. Final rinse with about 30 to 50 mL of methanol. Collect used methanol into an appropriate container for disposal.
15. After cleaning, wrap all the equipment apertures with aluminum foil.

**Ship the SPE column to the laboratory immediately. Elution from the SPE column must be completed within 7 days of extraction.**